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EVALUATION OF THE DCW 17 TREATMENT FOR MAGNESIUM ALLOYS

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F O R E W O R D

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EVALUATION OF THE DOW 17 TREATMENT FOR MAGNESIUM ALLOYS

SUMMARY

The operating parameters for the Dow 17 surface treatment of magnesium alloys have been determined. The thickness of Dow 17 coatings can be satisfactorily controlled from the thickness-time relationship at specific current densities.

The cleaning effect of Dow 17 has been compared with that of fluoride anodising. The results obtained were not consistent, but suggested that no significant deleterious effect would result from substituting a 1.0 mil thick Dow 17 treatment for fluoride anodising, chromic acid stripping and chromating.

Various thicknesses of Dow 17 pretreatment have been compared with standard DTD 911C pretreatment. The assessment included corrosion tests on the various pretreatments with surface sealing, painting to DTD 5580, and a full DTD 911C procedure. The results indicated that Dow 17 pretreatments in excess of 1.0 mil thick, were as good as the DTD 911C pretreatment.

The effect of various thicknesses of Dow 17 coatings on the fatigue strength of ZW3 and RZ5 alloys has been determined under rotating bending conditions. Significant reductions in the fatigue strength of ZW3 were observed with increasing thickness of Dow 17. However, comparable reductions were not observed with RZ5.

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1. INTRODUCTION

At the time of inception of the work described in this report (May 1968) the U.K. aircraft industry was increasingly undertaking joint projects with other European manufacturers, and an increasing number of American built, or American designed aircraft or components were being used. As a result, the Dow 17 process, widely used in the U.S.A. and Europe, was assuming increasing importance in the U.K.

Various users adopted different conditions for the Dow 17 treatment, and no comparative studies of their effectiveness as pretreatments had been made. In particular, no direct comparison had been made between Dow 17 pretreatments and fluoride anodising followed by stripping and chromating, as required in DTD 911C.

The purpose of this investigation was to study the Dow 17 process and evaluate the coating in comparison with equivalent currently used pretreatments. The following main areas were investigated:

(a) Investigation of operating parameters of the Dow 17 bath.

(b) Comparison of Dow 17 treatments with the DTD 911C required pretreatment.

(c) Comparison of various currently used protective systems based on Dow 17 with DTD 911C procedure.

(d) Comparison of fatigue properties after Dow 17 treatments.

Frequent reference is made throughout this report to the thickness of Dow 17 films. Film thicknesses were obtained by micrometer measurement, and are actually increases in dimension. The true film thicknesses would be somewhat greater due to inward growth of the film. The term "thickness" is used, however for simplicity.

2. INVESTIGATION OF OPERATING PARAMETERS OF DOW 17 BATH

2.1 INSTALLATION AND PREPARATION OF BATH

A Dow 17 pilot plant with an electrolyte capacity of 56 gallons was designed and installed. The plant consisted of a mild steel tank with external electrical heating coils (12 Kw) and a thermostat. As the tank could not be earthed during operation it was surrounded by a wooden guard, with removeable lid operating a safety cut-out on the anodising and heating circuits. A 40A auto-transformer supplied the anodising current via 1¼ ins. dia. aluminium bus bars to the electrode jigs. The electrode jigs were made in magnesium alloy after initial trials showed Dural jigs to cause variations in current density between panels on the positive and negative electrodes.

As the Dow 17 process operated in the temperature range 70 - 80^{o}C, and evolved HF fumes, evaporation from the surface of the bath was minimised by a layer of "Allplas" 20 mm. dia. hollow plastic spheres. Fume extraction equipment was also provided above the bath.

56 gallons of electrolyte were made up to the following formulation in tap water.

Ammonium Bifluoride (NH_4HF_2)	24% w/v
Sodium Dichromate ($Na_2Cr_2O_7.2H_2O$)	10% w/v
Phosphoric Acid (85% H_3PO_4)	9% v/v

2.2 EXPERIMENTAL DETAILS

2.2.1 Establishment of Parameters

ZW3 (BSL 504) alloy panels, dimensions 4 ins. x 2 ins. were used for initial trials to establish operating conditions. The composition of ZW3 and other alloys used in this evaluation are given in Table 1. The ZW3 panels were degreased, cleaned in boiling 10% chromic acid, then given a 10 second pickle in 3% nitric acid before anodising.

Panels were anodised in the range 20 to 50 A/ft^2 (calculated on the area of one electrode) using AC supply for times up to 30 mins. Voltages attained at the end of treatments were noted, and micrometer measurements on panels before and after treatment indicated the increase in dimensions due to anodising. The relationship between the various parameters was then determined.

2.3 RESULTS

2.3.1 Thickness of Coatings

The relationship between thickness and time could be interpreted as approximately linear up to 2 mils (0.002 ins.). For thicknesses in excess of 2 mils, particularly that produced at high current densities, the thickness increased more rapidly with time, (see Fig. 1). As would be expected, a greater film thickness was obtained for a given time with increasing current density.

The plot of thickness against terminal voltage in Fig. 2 showed that a rapid increase in thickness occurred at terminal voltages in excess of 100, particularly with higher current densities. Higher current densities gave higher terminal voltages for equivalent film build-up, although behaviour at 50 A/ft^2 was anomolous in this respect for lower film build-ups. (Fig. 2) No simple relationship between thickness, current density and terminal voltage could be seen.

The thickness of Dow 17 films up to approximately 1.5 mil, could with a little experience be very accurately judged from the colour of the films produced. The colour of both sides of each sample, and all samples from a batch of treated samples, was in every case similar, indicating that the throwing power of the treatment was exceptionally good.

2.3.2 Anodising of Test Panels

Having established a relationship between film build-up and anodising time at various current densities, batches of panels were anodised with specific thicknesses of Dow 17 for subsequent tests. Batches were anodised at 30 A/ft^2 on the basis of data shown in Figure 1. Actual thickness ranges obtained are shown in Figures 1 and 2. The thicknesses were slightly greater than predicted by the earlier tests, and in addition the terminal voltages were higher. These variations were probably due to changes in bath composition with increasing usage.

2.4 DISCUSSION

From the above results, the most promising method of controlling film thickness during anodising was by prediction from the thickness-time curves at specified current densities (Figure 1). Dow literature indicated that for a specified alloy, the total ampere minutes required was independent of current density. In Figure 2 thicknesses were plotted against total ampere minutes for all the trial runs carried out. Up to 1.5 mil, the relationship was approximately linear. However, some variation with current density was evident, and the use of the total ampere minute requirement alone to predict film thickness was not thought to be sufficiently valid.

When the initially determined film thickness-time relationship at 30 A/ft^2 was used to predict film thickness for subsequent batches of panels (2.3.2.), actual thicknesses produced were greater than those predicted. This was attributed to changes in bath composition with increasing usage. Subsequent anodising work carried out in the bath indicated that actual thickness had drifted to an even higher level than those shown in Figure 1. In order to compensate for this, the slope of the prediction line was increased empirically to the position indicated by a broken line in Figure 1. This was used successfully in later anodising work. It seems likely that parameters would have to be re-determined for a fresh bath or for a scaled up plant, but the present method has been found adequate for small scale work.

3. COMPARISON OF DOW 17 TREATMENTS WITH DTD 911C PRETREATMENT

3.1 INTRODUCTION

The purpose of this investigation was to compare various thicknesses of Dow 17 film with pretreatment in accordance with DTD 911C. The Dow 17 treatment had potential advantages in that, given good cleaning and corrosion resistant properties and compatability with stoved epoxy resins, the single process could replace the rather lengthy, and more expensive procedure of fluoride anodising, chromic acid stripping, activation and chromating. The investigation included the assessment of the cleaning effect produced by the application of various thicknesses of Dow 17 film in comparison with fluoride anodising and the comparsion of the corrosion resistance of surface sealed Dow 17 films with that of the current DTD 911C pretreatment.

3.2 COMPARISON OF THE CLEANING EFFECT OF THE DOW 17 TREATMENTS WITH DTD 911C PRETREATMENT

3.2.1 Introduction

Two separate tests were carried out. In the first the cleaning effect of a 1.0 mil Dow 17 film was compared with fluoride anodising on shot blasted A8 (BSL 122) alloy. In the second test ZRE1 (BSL 126) and ZW1 (BSL 507) alloys were used, and a range of Dow 17 and fluoride anodising treatments were applied.

3.2.2. Experimental Procuedure

Test 1

The investigation was carried out using a single ½ ins. thick cast A8 alloy plate, with a relatively smooth cast surface. 3 ins. x 3 ins. specimens were cut from the plate and heavily blasted on both surfaces with steel shot. Quadruplicate specimens were then given the following treatments;

1. Fluoride anodised 15 minutes at 120 v.

2. As (1) then stripped in boiling 10% chromic acid.

3. Dow 17 treated to give a nominal 1 mil per surface.

4. As (3) then stripped in boiling 10% chromic acid.

Specimens were weighed to ob[illegible] the increase in weight due to anodising, and re-weighed after stripping.

Corrosion testing was carried out in 3% sodium chloride solution saturated with $Mg(OH)_2$. Corrosion rates were determined after 76 and 414 hours.

Test 2

Four ZRE1 alloy cast plates, 8 ins. x 7 ins. x ½in. thick, and ½ in. thick rolled ZW1 plates were used. Both materials were heavily shot blasted with steel shot before cutting into 4 in. x 2 in. test panels. Panels were selected for treatment so that the whole range of pretreatments was carried out on panels from a single plate, in order to prevent inaccuracies resulting from differences between plates.

Panels from both alloys were given the following treatments:

(1) Untreated

(2) 0.5 mil Dow 17

(3) 1.0 mil Dow 17

(4) 1.5 mil Dow 17

(5) Fluoride anodised - 15 mins. at 120 volts.

(6) Fluoride anodised - 15 mins. at 90 volts.

Details of these treatments are given in Appendix I. Half the samples in each condition were then stripped in boiling 10% chromium trioxide solution.

Duplicate samples were weighed and immersed, in a 3% sodium chloride solution saturated with magnesium hydroxide. Corrosion rates were then determined after 168 hours.

3.2.3 Results

The results of corrosion tests are shown in Table 2.

In test 1, on A8 alloy specimens, the lowest corrosion rates were shown by specimens which had been fluoride anodised and stripped, followed by specimens Dow 17 treated and stripped. Specimens from which the films had not been removed showed higher corrosion rates, with Dow 17 treated specimens the highest. In test 2, on ZRE1 and ZW1 alloy specimens, the results were less decisive. The conclusions could be summarised as follows:

(1) The corrosion rate of ZRE1 was higher than that of ZW1 under all treatment conditions.

(2) Subsequent stripping of the anodised film made a major contribution to the reduction of corrosion rates after both Dow 17 and fluoride anodising, particularly with ZW1 samples.

(3) 120 volt fluoride anodising showed less cleaning effect than the 1.0 mil thick Dow 17 treatment, which was generally the most efficient cleaner of the Dow 17 treatments. Cleaning effects of 0.5 and 1.5 mil Dow 17 treatments were roughly comparable with that of the 120 volt fluoride anodising.

(4) 90 volt fluoride anodising gave less cleaning effect than the 120 volt fluoride anodising and was poorer than any of the Dow 17 treatments.

3.2.4 Discussion

The distinct superiority of fluoride anodising as a cleaning treatment observed in test 1 was not confirmed in test 2. The wider range of treatments indicated a greater variation in cleaning effect although both treatments had a considerable effect particularly after stripping.

The two equivalent practical processes under consideration were fluoride anodising followed by stripping in chromic acid, and Dow 17 without stripping. Both processes had a significant cleaning effect, although the relative merit varied from test 1 to test 2. In test 2 the best cleaning effect was given by a 1.0 mil Dow 17 film. Thicker or thinner films gave less cleaning effect.

In retrospect a more valid comparison would have been between a Dow 17 pretreatment and a pretreatment consisting of fluoride anodising, stripping in boiling chromic acid and then chrome manganese treatment. The effect of this would be to further improve the corrosion resistance of the fluoride anodised samples.

For all practical purposes, the results of both tests undertaken suggest that the cleaning effects obtained by Dow 17 treatment or by fluoride anodising and stripping are of the same order, and application of a Dow 17 pretreatment as a single process would not show serious disadvantages, from the cleaning aspect, compared to the 3 stage DTD 911C procedure.

3.3 COMPARISON OF THE PROTECTIVE VALUE OF DOW 17 TREATMENTS WITH DTD 911C PRETREATMENT

3.3.1 Material and Sample Treatment

The evaluation was carried out on sand cast high purity A8 (BSL 121) alloy plates and rolled sheet specimens in ZW3 (BSL 505). All sheet samples were cleaned by immersion in boiling chromic acid followed by a 10 second dip in 5% nitric acid. Batches of sheet samples were then given the following treatments:

(1) 0.5 mil build-up of Dow 17

(2) 0.8 mil build-up of Dow 17

(3) 1.3 mil build-up of Dow 17

(4) 1.5 mil build-up of Dow 17

(5) DTD 911C Pretreatment. (i.e. fluoride anodised, chromic acid stripped and 2 hours chrome manganese bath treatment)

Details of treatments are given in Appendix I. All samples were surface sealed with Araldite 985E resin. The coating produced a film thickness of 1.5 mil on the non-porous chrome manganese film and a correspondingly smaller build-up on the more porous Dow 17 films.

It was rather more difficult to obtain a predetermined thickness of Dow 17 film on the sand cast A8 samples, as accurate micrometer measurements could not be made on the rough sand cast surfaces, and no experience of anodising conditions for alloys other than ZW3 was available. Batches of shot blasted A8 alloy plates were therefore Dow 17 treated to give nominally thin, medium and thick coatings, by comparing the film weights obtained on A8 with those obtained on ZW3 alloy.

These samples were also surface sealed with Araldite 985E.

3.3.2 Evaluation Tests

Panels were subjected to the following tests:

Physical tests

1. Shot test

2. Bend test

Corrosion tests

1.	Salt Spray and Humidity Test	-	12 months
2.	R.A.E. Seawater Spray Test	-	12 months
3.	Atmospheric Exposure Test	-	12 months
4.	Distilled Water Column Test	-	12 months

Details of these tests are given in Appendix II.

3.3.3 Results

Physical Tests

The results of the bend and shot tests are given in Table 3. The adhesion of the Araldite 985E resin film to a chrome manganese film was better than that to the thin (0.5 - 0.8 mil) Dow 17 films. Thicker Dow 17 films tended to craze on the inside of the bend, and the resin was detached. Where this occurred part of the anodised film was removed with the resin, leaving, in the case of the thinner film, a very thin light buff coloured coating. With thicker films, a thin greenish-buff coloured film with minute areas of the darker green film remained.

The shot test also indicated this tendency for failure to occur within the Dow 17 film, rather than at the metal-film or film-resin interfaces. The superior adhesion of Araldite 985E to the chrome manganese films was again evident.

Corrosion Tests

Salt Spray and Humidity Test

Samples previously used for the shot test were used for this part of the evaluation. Details of examination after 12 months exposure are given in Table 4. On as cast A8 samples considerable asperity attack occurred on the thinner coatings. Medium and thicker Dow 17 coatings were less affected by asperity attack, and the thick Dow 17 film showed good resistance to creepage corrosion from all points of damage. DTD 911C procedure gave better protection than the thin or medium Dow 17 with surface sealing but thick Dow 17 with surface sealing showed the best protection.

On ZW3 panels, all breakdown was by creepage beneath the surface sealing resin and all the Dow 17 films gave better protection than DTD 911C procedure. The extent of creepage corrosion decreased with increasing thickness of Dow 17.

R.A.E. Seawater Spray Test

Details of the examination after 12 months exposure are given in Table 4.

The corrosion occurring during this test was more severe than that obtained in the humidity test although the amount of creepage corrosion was less. On cast panels, thin and medium Dow 17 films showed severe asperity attack, DTD 911C pretreatments showed slight asperity attack while the thick Dow 17 film was virtually unattacked. On rolled panels, the corrosion resistance of Dow 17 films increased with increasing thickness. DTD 911C treated panels were slightly worse than the thinnest Dow 17 treated panels.

Atmospheric Exposure Test

Little breakdown occurred on any of the panels although lightening in colour on asperities of cast panels could indicate slight asperity corrosion. This was apparent on all the cast panels, although slightly less marked on the DTD 911C treated panels. Rolled panels showed slight pitting along scribed crosses and sharp edges of coding, but there was no significant difference.

Distilled Water Column Test

All the as cast panels showed some electrical conduction shortly after the start of the test, but this did not increase significantly during the test. The DTD 911C treated panels showed slightly less conduction than the Dow 17 treated panels, but no true breakdown or corrosion occurred on any panels after 12 months.

3.3.4 Discussion

The inability of an unpigmented resin to adequately cover the asperities of cast surfaces was demonstrated by the Humidity and R.A.E. seawater spray tests.

The amount of creepage from points of damage decreased with increasing thickness of Dow 17 coating. The adhesion of surface sealing was better to the thicker, rather porous, Dow 17 coatings, than it was to the thinner, smoother and more compact Dow 17 coatings, where the resin was not able to fully impregnate the very fine pores.

On the smoother surface of DTD 911C treated rolled panels, the amount of creepage from points of damage was considerable. This was undoubtedly due to the reduced mechanical keying, compared to that obtained on a cast surface, and the resilient nature of the resin.

4. COMPARISON OF VARIOUS CURRENTLY USED PROTECTIVE SYSTEMS BASED ON DOW 17 WITH DTD 911C PROCEDURE

4.1 INTRODUCTION

The effect of Dow 17 pretreatment, as an alternative to DTD 911C procedure on the properties of the complete protective system was investigated. The coating system used was DTD 5580 with and without prior surface sealing. Dow 17 treatments by Procol in France and M.E.L. were investigated.

4.2 EVALUATION OF DOW 17 TREATMENT ON SIMPLE PANELS

4.2.1 Material and Sample Treatment

The evaluation was carried out on 3 ins. square by 6 swg. and 4 ins. x 2 ins. x 16 swg. rolled panels in ZW3 alloy. The panels were deburred, degreased and cleaned in 10% chromium trioxide solution followed by a 10 second dip in cold 5% nitric acid solution. Dow 17 treatment was carried out at 30 amps/sq. ft; the times being adjusted to give the required film thicknesses. Some samples were Dow 17 treated by Procol, France. These samples had a very thin (0.15 mil) greenish buff coloured film.

4.2.2 Protective Schemes Evaluated

Batches of panels were given the following surface treatments:

1. 0.15 mil Procol Dow 17 + Painting to DTD 5580.
2. 0.15 mil Procol Dow 17 + Surface Sealing + Painting to DTD 5580.
3. 0.5 mil Dow 17 + Painting to DTD 5580.
4. 0.5 mil Dow 17 + Surface Sealing + Painting to DTD 5580.
5. 0.8 mil Dow 17 + Painting to DTD 5580.
6. 0.8 mil Dow 17 + Surface Sealing + Painting to DTD 5580.
7. 1.7 mil Dow 17 + Painting to DTD 5580.
8. 1.7 mil Dow 17 + Surface Sealing + Painting to DTD 5580.
9. Fluoride Anodised, Stripped in boiling Chromic Acid, Chrome Manganese treated, Surface Sealed + Painting to DTD 5580.

Details of the treatments are given in Appendix I.

4.2.3 Evaluation Tests

Sample panels have been subjected to the following tests:

(a) Physical tests (bend tests, shot tests)

(b) Salt Spray - Humidity Test (Using shot test specimens) - 12 months

(c) R.A.E. Seawater Spray Test - 12 months

(d) Distilled Water Column Test - 15 months

The tests are fully described in Appendix II.

4.2.4 Results

(a) Physical Tests

The results of the shot and bend tests are given in Table 5. In all cases where failure occurred on the inside of the bend the underside of the detached paint or resin was green in colour, and some of the Dow 17 film could be seen adhering to it. A thin greenish-buff film remained on the metal surface. With the 0.3 mil Dow 17 film, very little could be seen adhering to the resin or paint, but with the thick film almost all the Dow 17 film was detached with the organic film.

Although the DTD 5580 system was sufficiently flexible to withstand bending to the point of metal failure around the outside of the bend when applied to a Dow 17 film it showed poor adhesion over the Araldite 985E resin film and either spalled or could be peeled off from the entire deformed area. No breakdown of the surface sealing occurred on the outside of the bend.

In the shot tests, panels with paint applied directly on the Dow 17 film spalled within the Dow 17 film except for very thin Procol Dow 17 treated panels, in which the failure appeared to be at the paint/Dow 17 interface. The poor adhesion of the DTD 5580 paint to surface sealing was again shown. There was no spalling of the surface sealing.

(b) Salt Spray-Humidity Test

The results of this test are given in Table 6. The thin Dow 17 treated panels which were not surface sealed showed severe creepage corrosion and some blistering. Protective schemes based on the thicker Dow 17 coatings had improved corrosion resistance. Although the adhesion of the DTD 5580 paint scheme to surface sealing was poor, the benefit of surface sealing was evident, even on the thin Dow 17 treated panels.

Medium and thick Dow 17 films with and without surface sealing gave better protection than the DTD 911C system but thin Dow 17 was inferior to DTD 911C, in both conditions. Best protection was given by the surface sealed medium (0.8 mil) and thick (1.7 mil) Dow 17 films.

(c) R.A.E. Seawater Spray Test

The results of this test are also shown in Table 6.

Comparatively little breakdown occurred. That which did occur was pitting at damage points with little creepage. Again medium and thick Dow 17 films gave good protection, but the thin films particularly without surface sealing were inferior to the DTD 911C procedure.

(d) Distilled Water Column Test

No apparent deterioration of any one of the schemes occurred during the 15 month exposure.

4.2.5 Discussion

All the tests showed an improvement in the protective value of Dow 17 films by surface sealing. In addition, the schemes based on thick and medium Dow 17 films with surface sealing were significantly superior to the DTD 911C scheme. However, the DTD 911C scheme was superior to the surface sealed thin Dow 17 scheme.

The DTD 5580 paint scheme showed poor adhesion to surface sealing and exhibited blistering under conditions of high humidity, particularly in the absence of surface sealing. This indicated some degree of water permeability in the polyurethane scheme.

4.3 EVALUATION OF DOW 17 TREATMENT ON FLANGE ASSEMBLIES

4.3.1 Material and Sample Treatment

The evaluation was carried out on standard M.E.L. flange assemblies. Each sand cast flange was approximately 4½" diameter and had a central boss, drilled and tapped to accommodate a nominally 1 ins. diameter pipe. Raised spot faces equally spaced around the flange were drilled to accommodate 5/16 ins. diameter bolts. Flanges were machined then given the appropriate protective treatment. Two such flanges, one in A8 alloy, and the other in ZRE1 alloy, were used in each assembly. They were bolted together using 5/16 ins. diameter BSF cadmium plated nuts, bolts and washers. A 3 ins. length of mild steel pipe was screwed into the boss of the A8 alloy flange and a similar length of chromated aluminium alloy (HT30WP) was screwed in the ZRE1 alloy flange.

4.3.2 Protective Schemes Evaluated

All the flanges were thoroughly deburred and degreased in trichloroethylene vapour. Batches of A8 and ZRE1 flanges were given the following treatment:

1. 0.15 mil Dow 17 (Procol)
2. 0.15 mil Dow 17 (Procol) + Surface Sealing
3. 0.8 mil Dow 17
4. 0.8 mil Dow 17 + Surface Sealing
5. 1.5 mil Dow 17
6. 1.5 mil Dow 17 + Surface Sealing

7. DTD 911C Pretreatment (Including Chrome Manganese)

8. DTD 911C Pretreatment (Including Chrome Manganese) + Surface Sealing

9. DTD 911C Pretreatment (Including Chrome Manganese) + J. Halls 588/0066

10. H.A.E.

11. H.A.E. + Surface Sealing

Details of these treatments are given in Appendix II.

The mild steel and aluminium alloy tubes were screwed into the flanges using Polycast Type 2 sealing compound. Additional Polycast was then used to caulk the magnesium/steel junction. Pairs of similarly treated flanges were bolted together; the nuts being tightened to a torque of 12 ft. lbs. Polycast was again used for assembly and caulking the joints and bolts.

All flange assemblies with the exception of those having the J. Halls 586/0066 coating were then painted to DTD 5580. Those having the J. Halls treatment were painted to DTD 5555. Details of the paint treatments are given in Appendix I.

4.3.3 Corrosion Tests

Assemblies were subjected to the following tests.

(a) Salt Spray and Humidity - 1 year

(b) R.A.E. Seawater Spray - 1 year

(c) Marine Atmospheric Exposure (Beaumaris) - 2½ years

Details of these tests are given in Appendix II.

4.3.4 Results

(a) Salt Spray and Humidity Test

Details of the examination of the assemblies after 12 months exposure are summarised in Table 7. The assemblies have been given a corrosion rating, A to E, based on visual assessment of the degree of blister and breakdown. All the assemblies which had been overcoated with DTD 5580 paint scheme showed blistering to some extent. Corrosion breakdown had occurred beneath some blisters, particularly adjacent to spot faces and on flange edges. Blistering and breakdowns were more pronounced on non-surface sealed flanges as can be seen by comparing Figures 4 and 5 which show the maximum blistering observed, and the improvement produced by surface sealing. Fewer blisters were evident on the H.A.E. treated assembly.

The flange assembly coated with J. Halls system and overcoated with DTD 5555 showed no blistering, but some breakdown had occurred.

All the non-surface sealed assemblies showed poor resistance to high humidity conditions. Systems incorporating surface sealing showed a higher order of corrosion protection. The best protection was given by H.A.E., surface sealed and overpainted with DTD 5580, which showed no breakdown and only minute localised blistering. The standard DTD 911C system fell into the same general category as the other systems embodying surface sealing.

(b) R.A.E. Seawater Spray Test

Table 7 summarises the results of 12 months exposure. A corrosion rating has again been given. Severe corrosion breakdowns occurred on all assemblies which had not been surface sealed. The largest and most numerous breakdowns occurred on flanges with the very thin Procol Dow 17 pretreatment (Fig. 6) and became less severe with increasing thickness of Dow 17 film. No significant breakdown occurred on any flange assemblies which had been surface sealed, although single isolated points of breakdown were observed on the assemblies with 0.15 (Fig. 7) and 0.8 mil Dow 17 coatings. 1.5 mil Dow 17, H.A.E. and DTD 911C pretreatments with DTD 5580 top coats all showed no breakdown.

The extent of breakdown was generally more severe on A8 than on ZRE1 flanges. The J. Hall's 588/0066 plus DTD 5555 system showed breakdown on the A8 flange, although the corresponding ZRE1 flange showed none. The severe blistering which occurred with DTD 5580 coated assemblies in the humidity test did not occur in the R.A.E. test.

(c) Marine Atmospheric Exposure

Flange assemblies were examined after 2½ years exposure. The A8 alloy flange of the 0.15 mil Dow 17 (Procol) plus DTD 5580 treated assembly had three corrosion creepages. None of the other assemblies showed any blistering or breakdowns.

Slight degradation of the paint itself was evident, as shown by cracking of the DTD 5580 and DTD 5555 paints on the corners of the steel washers etc. The epoxy finish on the J. Halls 588/0066 plus painting to DTD 5555 scheme had chalked considerably.

4.3.5 Discussion

The results of the Humidity and R.A.E. Seawater Spray Tests indicated in a most emphatic manner the necessity to surface seal components operating in corrosive environments. The poor corrosion resistance of all the pretreatments examined without surface sealing may be due to the water permeability and poor adhesion of the DTD 5580 system, as indicated by the severe blistering occurring in the humidity test. As would be expected, increase in thickness of anodic coating improved corrosion resistance, although it did not itself prevent corrosion, even in the case of the H.A.E. film. Surface sealing prevented significant corrosion on all the pretreatments studied and no real differentiation could be made between the various systems although earlier work (3.4.2) on simple panels indicated that thinner Dow 17 films (up to 0.5 mil) were inferior to DTD 911C pretreatment even when surface sealed.

The marine atmospheric exposure test at Beaumaris proved to be too mild a corrosive environment to be useful in the evaluation of complete protective systems.

5. COMPARISON OF THE FATIGUE PROPERTIES OF DOW 17 TREATMENTS

5.1 INTRODUCTION

The object of the investigation was to determine the effect of varying thicknesses of Dow 17 on the fatigue strengths of RZ5 and ZW3 alloys, using an Avery 6302 Wohler-type fatigue testing machine. The S/N curves obtained would be comparable with those previously obtained for the same alloys with a wide range of surface treatments.[1]

5.2 MATERIAL AND SAMPLE TREATMENT

50 sand cast RZ5 (L 128) alloy DTD bars were produced and machined to standard Avery type, Wohler fatigue bars with a gauge diameter of 0.2629". 40 ft. of 1 ins. diameter extruded bar in ZW3 (L 505) alloy was also produced. The chemical compositions and mechanical properties of the alloys are given in Table 8. Stringent precautions were taken to obtain lengths of extrusion free of defect, and these were then machined to fatigue bars. The fatigue bars in both alloys were randomised prior to surface treatment to avoid any progressive deviations arising from factors associated with casting or extrusion of the material.

Batches of 12 fatigue bars from each alloy were Dow 17 treated to produce a film build-up of 0.5, 1.0 and 1.5 mils. The test area only was anodised; the remainder was blanked off during anodising. Thickness of film produced by anodising was obtained by micrometer measurement at the point of maximum stress before and during treatment until the desired thickness was achieved. Bars were also retained in the un-anodised condition for comparison.

5.3 RESULTS

S/N curves for plain pumiced, thin (0.5 mil), medium (1.0 mil) and thick (1.5 mil) Dow 17 treated specimens in Heat Treated RZ5 (BSL 128) and extruded ZW3 (BSL 505) have been obtained. The results are plotted in Figs. 8 and 9. The curves show that Dow 17 treatments had no significant effect on the fatigue properties of RZ5 alloy. On the other hand, a marked reduction in the fatigue properties of ZW3 was produced. The effect varied from a reduction in fatigue strength of approximately 9% for the 0.5 mil Dow 17 coating to 21% for the 1.5 mil thick Dow 17 coating at 5×10^7 cycles.

Spalling of the anodic coating occurred on fracture with all three Dow 17 coatings on ZW3 but no such spalling occurred on RZ5.

Examination of metallographic sections taken from the fractured fatigue bars showed that the Dow 17 treatments produced a roughening of the metal surface; the roughening increased with increasing film thickness. There was no discernable difference in the surface roughness produced by similar Dow 17 treatments on RZ5 and ZW3.

5.4 DISCUSSION

It is known that treatments resulting in pitting or roughening of a metal surface result in the reduction in the fatigue strengths of the material, and that the greater the roughening effect, the greater the reduction in fatigue strength. Since any Dow 17 treatment of magnesium produces some degree of surface roughening, the treatment is likely to reduce the fatigue properties of magnesium alloys. Although this was shown to be the case with ZW3 alloy when reductions in fatigue strengths of up to 21% were obtained, the effect of Dow 17 treatments on RZ5 was negligible.

The notch sensitivity of magnesium casting alloys is generally lower than that of wrought alloys. Consequently the effect of Dow 17 treatments on the fatigue strength of RZ5 should be less than that on ZW3. However, some reduction in the fatigue strength of F[illegible] would be expected. It was not possible to explain this apparent anomoly from the results and observations made.

A comparison of these results with those of previous work[1] showed that the effect of Dow 17 and H.A.E. anodising on the fatigue properties of ZW3 was, thickness for thickness, very similar. The reduction in fatigue properties of ZW3 alloy resulting from a 0.5 mil thick Dow 17 treatment was similar to that produced by a chrome manganese treatment. However, the chrome manganese treatment had no significant effect on the fatgiue properties of RZ5 alloy.

6. GENERAL CONCLUSIONS

The following conclusions can be drawn from the results of the work.

INVESTIGATION OF OPERATING PARAMETERS OF DOW 17 BATH

1. It is possible to control the thickness of Dow 17 film with a reasonable degree of accuracy by controlling the anodising time at specific current densities in accordance with the graphs shown in Fig. 1.

COMPARISON OF DOW 17 TREATMENTS WITH DTD 911C PRETREATMENT

1. No significant deleterious effects would result from substituting a 1.0 mil thick Dow 17 treatment for fluoride anodising.

2. 1.0 mil, and thicker, Dow 17 coatings are comparable to the DTD 911C pretreatment as bases for further organic protection e.g. surface sealing. Thinner coatings, particularly the very thin coatings commonly used in the U.S.A. and Europe, are inferior to DTD 911C procedure, and their use is not recommended.

3. Dow 17 coatings have been shown to be porous and consequently must be surface sealed for optimum protection.

COMPARISON OF VARIOUS CURRENTLY USED PROTECTIVE SYSTEMS BASED ON DOW 17 WITH DTD 911C PROCEDURE

1. Systems based on surface sealed Dow 17 coatings were comparable with DTD 911C procedure. Dow 17 films, without surface sealing were inferior to DTD 911C.

2. The use of wet assembly techniques were essential to avoid the galvanic corrosion of magnesium in complex assemblies.

3. The DTD 5580 paint scheme exhibited blistering under high humidity test conditions indicating that the paint was to some extent water permeable.

COMPARISON OF THE FATIGUE PROPERTIES OF DOW 17 TREATMENTS

1. The fatigue strength of ZW3 was considerably reduced by Dow 17 treatments. No similar deleterious effect was obtained with RZ5 and further work is required to explain this apparent anomoly.

7. REFERENCES

1. Emley, E.F. Principles of Magnesium Technology, 657 - 660
Pergamon Press (1966)

TABLE 1: CHEMICAL COMPOSITIONS OF THE MAGNESIUM ALLOYS USED IN THE EVALUATION OF THE DOW 17 TREATMENT

ALLOY	British Standard Specification	Alloying Elements (%)				
		Zinc	Zirconium	Aluminium	Manganese	Rare Earth Metals
Z W 3	L 504/5	3.0	0.6			
Z W 1	L 507	1.3	0.6			
R Z 5	L 128	4.0	0.7			1.2
Z R E 1	L 126	2.2	0.6			2.7
A 8	L 121	0.5		8.0	0.3	

TABLE 2: RESULTS OF TESTS TO COMPARE CLEANING EFFECT OF DOW 17 TREATMENTS WITH FLUORIDE ANODISING

Section 3.2

Test 1

Alloy	Treatment	Average Corrosion Rate * ($mgs/cm^2/day$) After 76 hrs.		Average Corrosion Rate ($mgs/cm^2/day$) After 414 hrs.	
		Unstripped	Stripped in CrO_3	Unstripped	Stripped in CrO_3
A 8	120 v Fluoride Anodise	3.74	1.51	5.76	3.10
	1 mil Dow 17	5.34	2.14	9.33	4.04

Test 2

Alloy	Treatment	Average Corrosion Rate ($mgs/cm^2/day$) After 168 Hours	
		Unstripped	Stripped in CrO_3
Z R E 1	None	39.3	43.0
	0.5 mil Dow 17	32.2	16.1
	1.0 mil Dow 17	10.5	15.1
	1.5 mil Dow 17	16.9	18.2
	120 v Fluoride Anodising	30.3	18.5
	90 v Fluoride Anodising	34.8	21.4
Z W 1	None	> 46	14.3
	0.5 mil Dow 17	9.2	0.4
	1.0 mil Dow 17	3.2	0.7
	1.5 mil Dow 17	8.8	1.0
	120 v Fluoride Anodising	7.9	5.7
	90 v Fluoride Anodising	28.1	10.7 **

* Specimens washed and scrubbed only. Not cleaned in chromic acid

** Wide Spread in corrosion rates.

TABLE 3: RESULTS OF BEND AND SHOT TEST

Surface Treatment	Result of Bend Test		Result of Shot Test*	
	Outside	Inside	Front Face	Rear Face
0.5 mil Dow 17 + Surface Seal	Detachment of resin along 1" of fracture to 1/16" max.	Detachment of resin along whole break to ¼" max.	Detachment of resin along shock lines from shot up to 5/16" max.	Detachment of resin up to 5/16" from metal fracture
0.8 mil Dow 17 + Surface Seal	No apparent breakdown	Detachment of resin along whole break to 5/16" max.	No apparent breakdown	Detachment of resin up to ⅛" from metal fracture
1.3 mil Dow 17 +	No apparent breakdown	Detachment of resin along whole break to 3/16" max. Further crazing to 5/16"	Detachment of resin along shock lines from shot up to 7/16"	Detachment of resin up to ⅛" from metal fracture
1.5 mil Dow 17 + Surface Seal	No apparent breakdown	Detachment of resin along whole break to 5/32" max. Further crazing to 5/16"	Detachment of resin along shock lines from shot up to ¼" max.	Detachment of resin up to ⅛" from metal fracture
Chrome Manganese + Surface Seal	No apparent breakdown	Detachment of resin along whole break to ⅛" max.	No apparent breakdown	No apparent breakdown

* The shot test carried out on cast material resulted in cracking or breaking of the specimens, with no further breakdown of the coatings.

TABLE 4: RESULTS OF 12 MONTHS HUMIDITY TEST AND 12 MONTHS R.A.E. SEAWATER SPRAY TEST ON VARIOUS THICKNESSES OF DOW 17 FILM WITH SURFACE SEALING

Section 3.3

Material	Surface Treatment	12 Months Shot, Salt Spray Humidity Test				12 Months R.A.E. Seawater Spray		
		No.	Creepage from Cross	Creepage from shot	General Surfaces	No.	Creepage from Cross	General Surfaces
HP.A8 Cast Panels	Dow 17 (0.46 oz/yd^2) + Surface sealing	1) 2)	General asperity breakdown on front face with creepage. Creepage from shot, and cracks on rear up to 10 mms. Creepage from shot, cross, cracks to 10 mms. on frontface			3) 4)	General heavy corrosion up to 15 mms.	Numerous breakdown on both surfaces up to 15 mms.
	Dow 17 (0.94 oz/yd^2) + Surface sealing	9	Large areas to 15 mms	General to 16 mms.	Asperity b.d. around shot to 10 mm.	11	Pitting up to 8 mms. max.	Numerous b.d. on both faces to 20 mms. max. Generally $<$ 8 mm
		10	Few to 1 mm.	3 mms. max.	Asperity b.d. at shot to 10 mm. max. Creepage from cracks on rear.	12	Pitting to 5 mms max.	Several b.d. on both sides to 7 mms. max.
	Dow 17 (1.53 oz/yd^2) + Surface sealing	17	$<$ 1 mm.	$<$ 1 mm.	Slight asperity b.d. near shot. Creepage from cracks on rear face to 13 mms.	19	4 b.d. $<$ 1 mm.	4 b.d. on rear face to 4 mm. max
		18	$<$ 1 mm.	$<$ 1 mm.	Slight asperity b.d. near shot. Creepage from cracks on rear face to 5 mm.	20	4 b.d. $<$ 1 mm.	Several b.d. on both faces. $<$ 1 mm.
	Fluoride Anodised + CrO_3 + Chrome Manganese + Surface Sealing	25	6 to 3 mms.	$<$ 3 mms.	Slight asperity b.d. on front face only.	27	1 b.d. $<$ 1 mm.	Numerous asperity b.d. on both face $<$ 1 mm.
		26	4 to 1 mm. max.	$<$ 3 mms.	Slight asperity b.d. on front face only.	28	6 b.d. $<$ 1 mm.	Several asperity b.d. on both face to 5 mms. max. Numerous $<$ 1 mms.
	Dow 17 (0.5 mil) + Surface sealing	65	8 to 13 mms. max.	18 mms. max.	Creepage from edge on rear face to 13 mms. 7 creepages on rear face to 23 mms.	67	24 b.d. to 4 mms max.	b.d. around hole coding to 3 mms.
		66	12 to 12 mms. max.	17 mms. max.	7 creepage on rear face to 23 mms.	68	30 b.d. to 3 mms max.	b.d. around hole, coding to 3 mms.
ZW3 Rolled Panels	Dow 17 (0.8 mil) + Surface sealing	33	Several $<$ 0.5 mms.	11 mms. max.	Creepage from damage at rear to 4 mms, and from edge to 6 mms.	35	10 b.d. $<$ 1 mm.	V. slight b.d. at holes and coding.
		34	6 to 11 mms. max.	19 mms. max.	Creepage from damage at rear to 7 mms.	36	10 b.d. $<$ 1 mm. 1 to 2 mms.	Slight b.d. at holes, coding to 1 mm. max.
	Dow 17 (1.3 mil) + Surface sealing	41	Several $<$ 0.5 mms.	7 mms. max.	Creepage from damage at rear to 7 mms.	43	7 b.d. $<$ 1 mm.	Slight b.d. at holes, coding to 1 mm. max.
		42	1 to 5 mms. 1 to 1mm	11 mms. max.	Creepage from damage at rear to 8 mms.	44	7 b.d. $<$ 1 mm.	As for 43
	Dow 17 (1.5 mil) + Surface sealing	49	3 to 1 mm. max.	14 mms. max.	Creepage from damage at rear to 4 mms.	51	20 b.d. $<$ 1 mm.	Slight b.d. at holes, coding edges to 1 mm. max.
		50	4 to 2 mms. max.	14 mms. max.	Creepage from damage at rear to 3 mms.	52	10 b.d. to 1 mm max.	As for 51
	Fluoride Anodised + CrO_3 + Chrome Manganese + Surface sealing	57	Numerous to 14 mms. max.	23 mms. max.	Creepage from damage at rear to 13 mms. and from edge to 10 mms.	59	Numerous to 3 mm max.	b.d. at holes, edges coding to 2 mms. One b.d. on front face 2 mms.
		58	Numerous to 19 mms. max.	33 mms. max.	Creepage from damage at rear to 10 mms. Several creepages on front to 13 mms.	60	Numerous to 5 mm max.	B.d. at holes, edges, coding to 2 mms. One b.d. on rear face 4 mm.

TABLE 5: RESULTS OF BEND AND SHOT TESTS ON VARIOUS THICKNESSES OF DOW 17 FILM WITH AND WITHOUT SURFACE SEALING PLUS PAINTING TO DTD 5580

Section 4.2

Sample Treatment	RESULTS OF BEND TESTS				RESULTS OF SHOT TESTS	
	Inside bend		Outside Bend			
	Description	Failure	Description	Failure	Front Face	Rear Face
0.15 mil Dow 17 + DTD 5580	N.D.		N.D.		Spalling of paint up to 9 mms.	Spalling of paint up to 9 mms.
0.15 mil Dow 17 + S.S. + DTD 5580	N.D.		N.D.		Spalling of paint and surface sealing up to 12 mms.	Spalling of paint and surface sealing up to 6 mms.
0.5 mil Dow 17 + DTD 5580	Detachment of paint to 6 mms. max.	Dow 17 /paint	No breakdown	-	Spalling of paint to 12 mm	Spalling of paint to 12 mm
0.5 mil Dow 17 + S.S. + DTD 5580	Detachment of total film up to 6 mms. max.	Dow 17 / S.S.	Detachment of paint only up to 8 mms.	S.S./ paint	Spalling of paint only to 50 mms.	Spalling of paint to 4 mms. Cracking of paint to 25 mms.
0.8 mil Dow 17 + DTD 5580	Detachment of paint + some Dow 17 to 8 mms.	In Dow 17	No breakdown		Spalling of paint to 15 mms.	Spalling of paint to 4 mms. Further cracking of paint to 9 mms.
0.8 mil Dow 17 + S.S. + DTD 5580	Detachment of paint S.S. and some Dow 17 to 8 mms.	In Dow 17	Detachment of paint only up to 9 mms.	S.S./ piant	Spalling of paint only to 40 mms.	Spalling of paint only to 25 mms. + spalling of S.S. to 3 mms. from cracks
1.7 mil Dow 17 + DTD 5580	Detachment of paint + some Dow 17 to 8 mms.	In Dow 17	No breakdown	-	Spalling of paint to 25 mms.	Spalling of paint to 4 mms. Further cracking of paint to 12 mms.
1.7 mil Dow 17 + S.S. + DTD 5580	Detachment of paint, S.S. and some Dow 17 to 5 mms. Further cracking to 6 mms.	In Dow 17	Detachment of paint only to 9 mms.	S.S./ paint	Spalling of paint only to 38 mms. some crazing of exposed S.S.	Spalling of paint only to 25 mms. Further cracking to 12 mms. Crazing and spalling of S.S. to 4 mms.
Chrome Manganese + S.S. + DTD 5580	Detachment of paint + S.S. to 2 mms. max.	Chromate/ S.S.	Detachment of paint only to 9 mms.	S.S./ paint	Spalling of paint only to 15 mms. generally but 43 mms. along cracks	Spalling of paint only to 4 mms. Further cracking of paint to 25 mms.

S.S. = Surface Sealed with Araldite 985E

TABLE 6: RESULTS OF 12 MONTHS HUMIDITY TEST AND 12 MONTH R.A.E. SEAWATER SPRAY TEST ON VARIOUS THICKNESSES OF DOW 17 FILM WITH AND WITHOUT SURFACE SEALING PLUS PAINT TO DTD 5580

Sample Treatment	12 Months Shot, Salt Spray + Humidity Test				12 Months R.A.E. Seawater Spray	
	Creepage from Cross	Creepage from Shot Front	Creepage from Shot Rear	General Surfaces	Creepage from Cross	General Surfaces
0.15 mil Dow 17 + DTD 5580	3 up to 15 mms. + 1 up to 7 mms.	5 mms.	8 mms.	Numerous blisters 0.5 mms.	12 up to 3 mms.	No breakdown
0.15 mil Dow 17 + S.S. + DTD 5580	3 up to 11 mms.	12 mms.	12 mms.	No breakdown	12 up to 3 mms.	No breakdown
0.5 mil Dow 17 + DTD 5580	-	37 mms.	-	Creepage from damaged edges to 22 mms. Numerous minute blisters	21 to 5 mms.	No breakdown
	10 up to 15 mms. max. + numerous 0.5 mms.	-	-	Creepage from bottom hole to 17 mms.	15 to 5 mms.	No breakdown
0.5 mil Dow 17 + S.S. + DTD 5580	-	30 mms.	18 mms.	No breakdown	8 b.d. to 2.5 mms. max.	No breakdown
	10 to 25 mms. max.	-	-	No breakdown	18 b.d. to 2.5 mms. max.	No breakdown
0.8 mil Dow 17 + DTD 5580	-	1 mm.	10 mms.	Corrosion of exposed Dow 17 film. Creepage from bottom hole to 17 mms	4 to 2 mms. + 10 0.5 mms.	No breakdown
	10 to 15 mms. max. + several to 1 mm.	-	-	No breakdown	6 to 2 mms. + 6 to 1 mm.	No breakdown
0.8 mil Dow 17 + S.S. + DTD 5580	-	16 mms.	5 mms.	No breakdown	Breakdown along part of cross to 1.5 mms.	No breakdown
	1 to 4 mms. + 5 to 1.5 mms.	-	-	No breakdown	b.d. at several points to 1 mm.	No breakdown
1.7 mil Dow 17 + DTD 5580	-	10 mms.	-	Slight creepage from damaged edge	5 to 1.5 mms. max.	No breakdown
	6 to 1 mm.	-	-	No breakdown	6 to 1 mm. max.	No breakdown
1.7 mil Dow 17 + S.S. + DTD 5580	-	15 mms.	8 mms.	No breakdown	b.d. at several points to 1.5 mms.	No breakdown
	3 to 1 mm. max	-	-	No breakdown	b.d. at several points to 1.5 mms.	No breakdown
Fluoride Anodised + CrO_3 strip + Chrome Manganese + S.S. + DTD 5580	-	18 mms.	-	No breakdown	8 to 2 mms. max.	No breakdown
	8 to 2 mms. max.	-	-	Slight blistering along edges	9 to 2 mms. max.	No breakdown

S.S. = Surface Sealing with Araldite 985E

TABLE 7: RESULTS OF 12 MONTHS HUMIDITY TEST AND 12 MONTHS R.A.E. SEAWATER SPRAY TESTS ON FLANGE ASSEMBLIES

Section 4.3

Surface Treatment	12 Month Salt Spray-Humidity Test		12 Month R.A.E. Seawater Spray Test	
	No.	C.R.*	No.	C.R.*
0.15 mil Dow 17 (Procol) + DTD 5580	2809 2/8	D	2809 1/7	E
0.15 mil Dow 17 (Procol) + Surface Sealing + DTD 5580	2809 5/11	B	2809 4/10	B
0.8 mil Dow 17 + DTD 5580	2848 38/62	D	2848 37/61	E
0.8 mil Dow 17 + Surface Sealing + DTD 5580	2848 41/65	C	2848 40/64	C
1.5 mil Dow 17 + DTD 5580	2848 44/56	D	2848 43/55	D
1.5 mil Dow 17 + Surface Sealing + DTD 5580	2848 47/59	B	2848 46/58	A
Fluoride Anodised, Stripped, Chrome Manganese + Surface Sealing + DTD 5580	2848 50/68	C	2848 49/67	A
Fluoride Anodised, Chrome-Manganese + DTD 5580	2848 53/71	D	2848 52/70	E
Fluoride Anodised, Stripped, Chrome-Manganese + J. Halls 588/0066 + DTD 5555	2866 2/4	C	2866 1/13	E
H.A.E. Anodised + DTD 5580	2866 5/17	D	2866 4/16	E
H.A.E. Anodised + Surface Sealing + DTD 5580	2866 8/21	A	2866 7/20	B

*C.R. = Corrosion Rating as follows:

A No significant deterioration

B No visible corrosion. Slight Blistering only

C Small isolated corrosion breakdowns only with slight blistering

D More general corrosion breakdown and/or general blistering

TABLE 8: CHEMICAL COMPOSITIONS AND TENSILE PROPERTIES OF ALLOYS USED FOR FATIGUE TESTS

Section 5

Alloy	B.S. Specification	Chemical Composition			Tensile Properties			
		Zinc	Zirconium	Rare Earth	0.1% Proof Stress (t.s.i.)	0.2 Proof Stress (t.s.i.)	Ultimate Tensile Stress (t.s.i.)	Elongation on $4\sqrt{A}$
R Z 5	L 128	4.44	0.70	1.26	7.9	8.5	12.7	3.4%
Z W 3	L 505	2.74	0.54	-	14.7	16.1	20.4	19 %

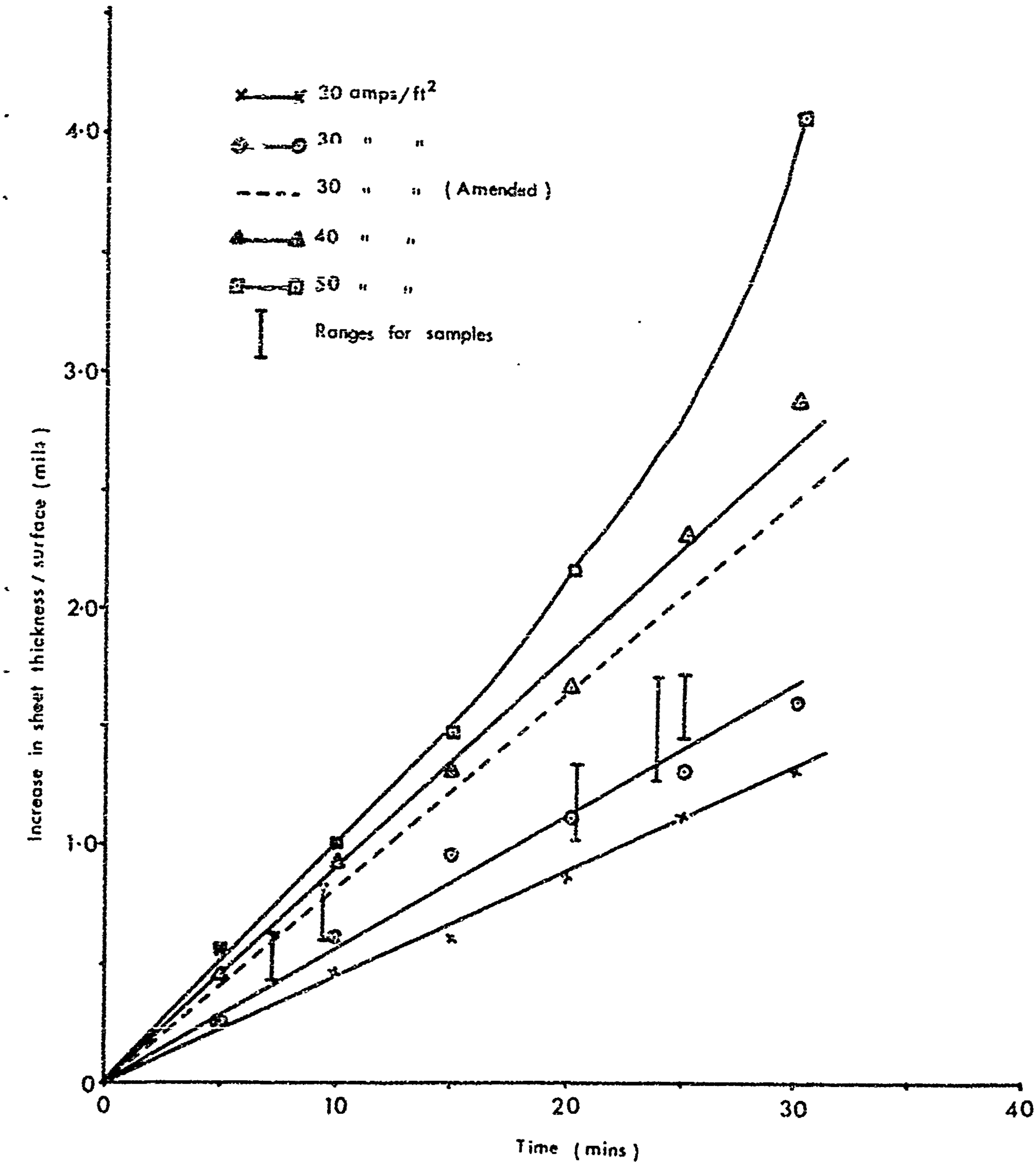


Fig: 1. Increase in Sheet Thickness/Surface v Time for Dow 17 treatment at various current densities.

Section 2.

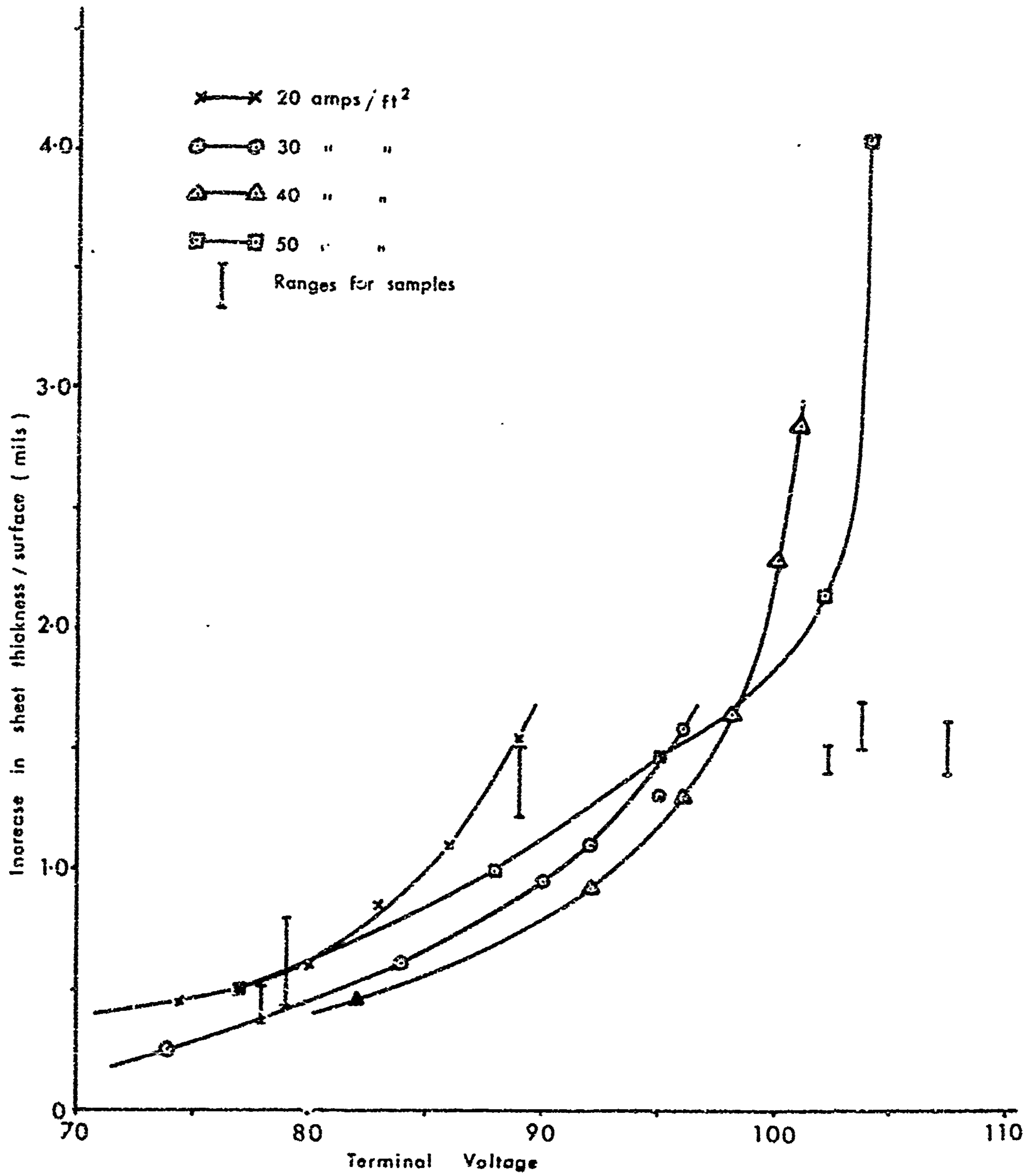


Fig. 2: Increase in Sheet Thickness/Surface v Terminal Voltage for Dow 17 Treatment at Various Current Densities.

Section 2.

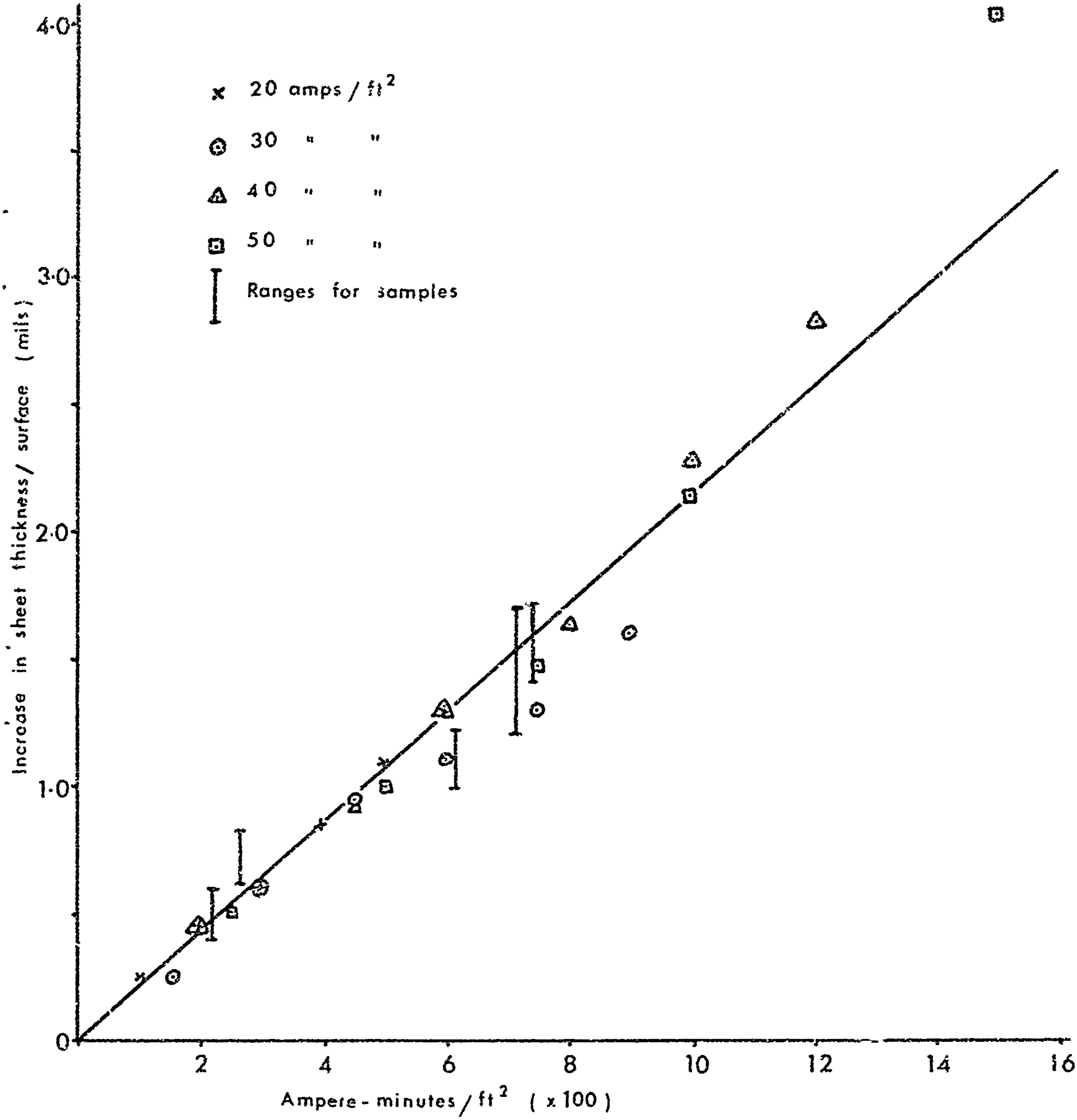


Fig. 3: Increase in Sheet Thickness/Surface v Total Ampere Minutes at Various Current Densities.

Section 2.

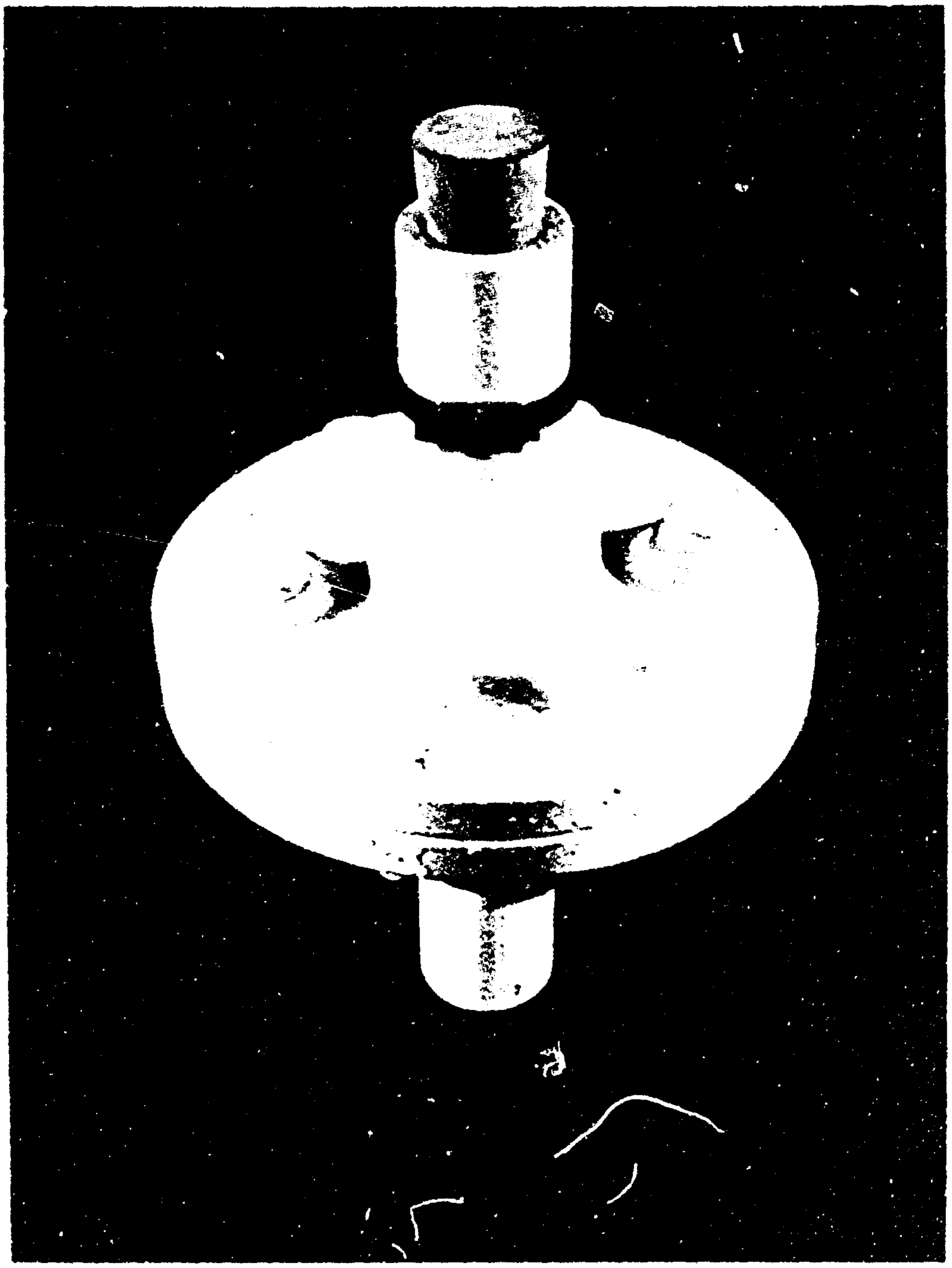


Fig. 4: ZRE1 Flange After 12 months Humidity Test.
Procol Dow 17 + DTD 5580 Paint Scheme. Section 1.5

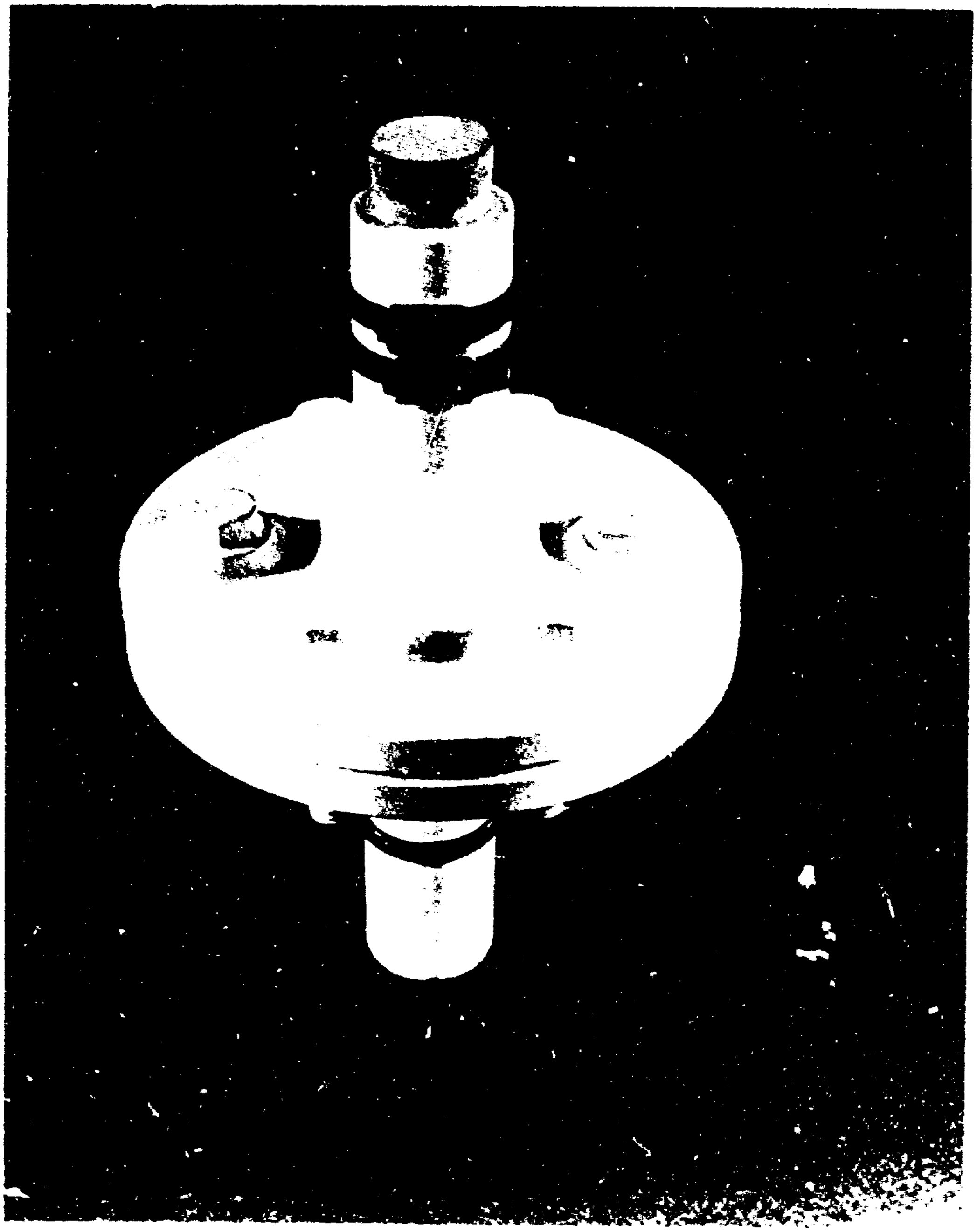


Fig. 5: ZRE1 Flange After 12 Months Humidity Test.
Procol Dow 17 + Surface Sealing + DTD 5580 Paint Scheme
Section 4.3.

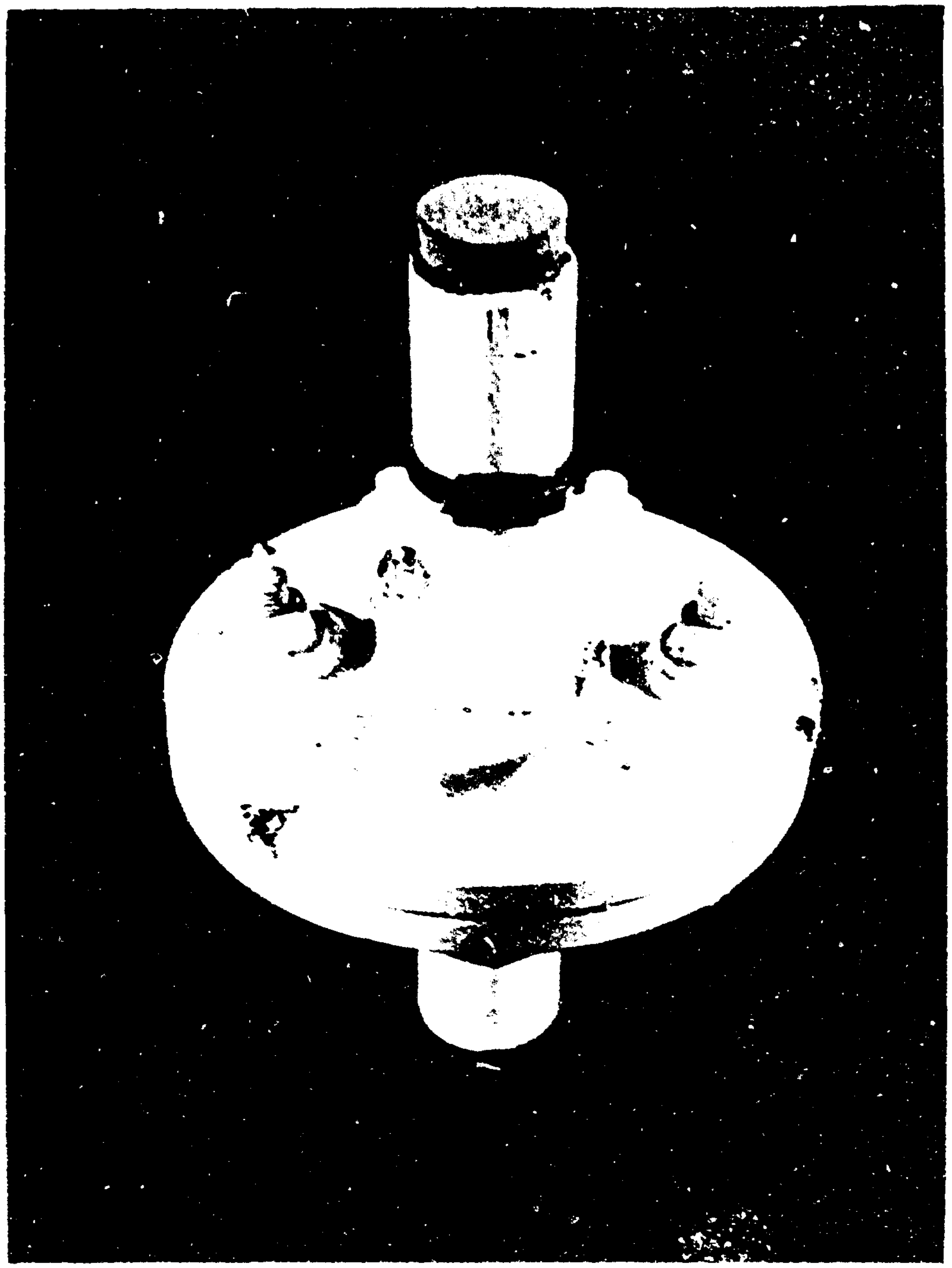


Fig. 6: A8 Flange After 12 Months R.A.E. Test.
Procol Dow 17 - DTD 5555 Paint Scheme. Section 4.3

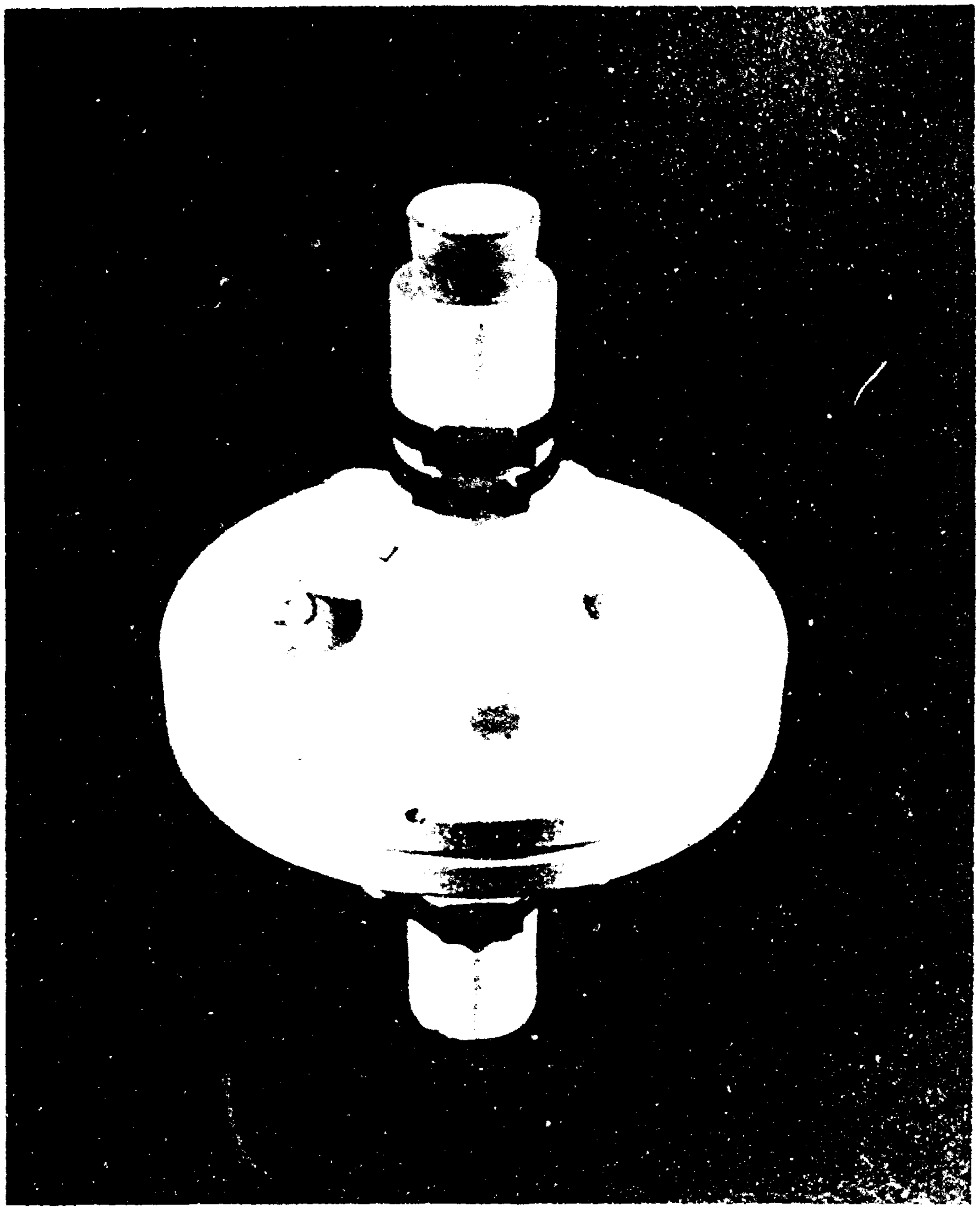


Fig. 7: ZRE1 Flange After 12 Months R.A.E. Test.
Procol Dow 17 + Surface Sealing + DTD 5580 Paint Scheme,
Showing Isolated Breakdown. Section 4.3

FIGURE 8 ROTATING BENDING FATIGUE TEST

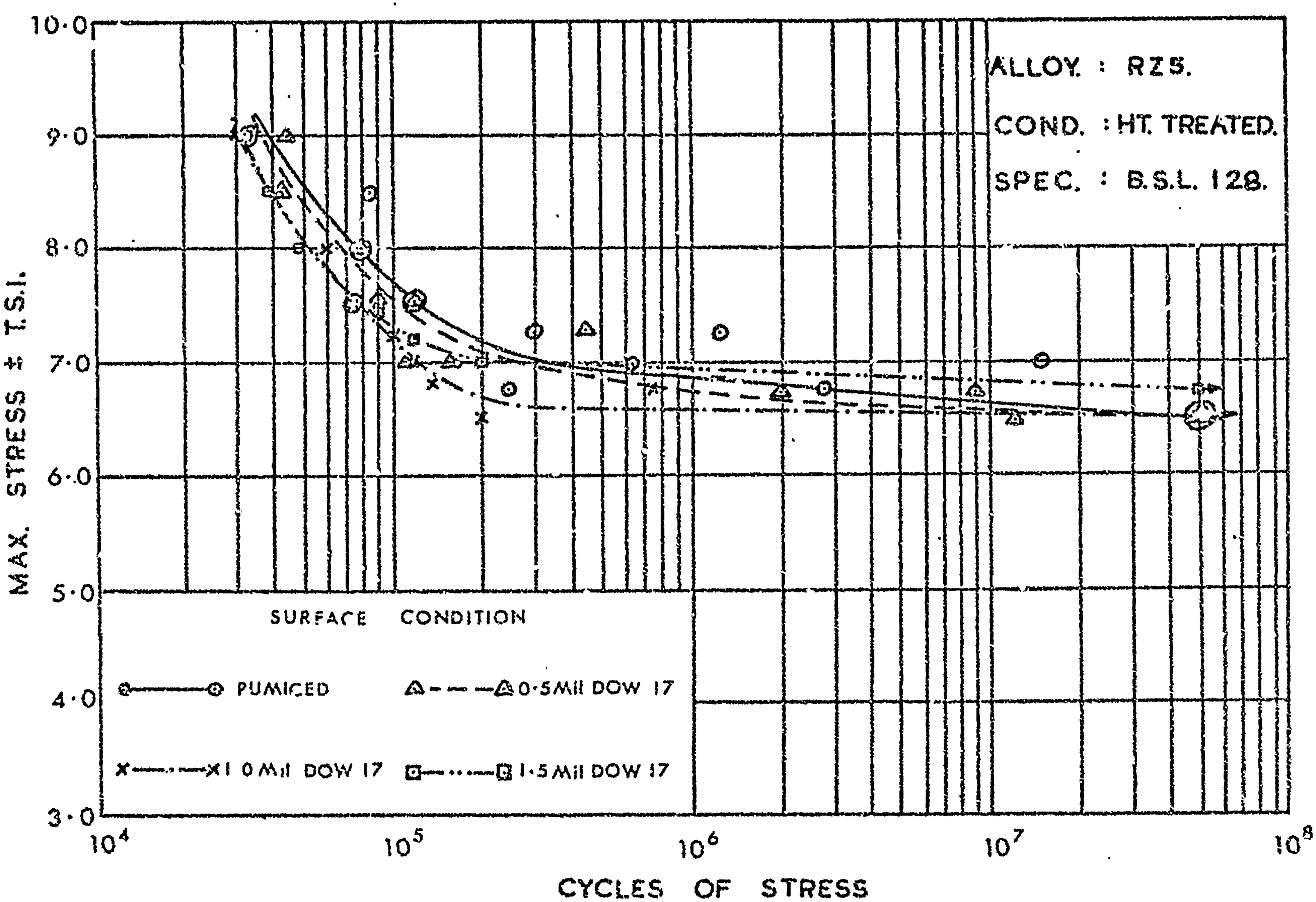
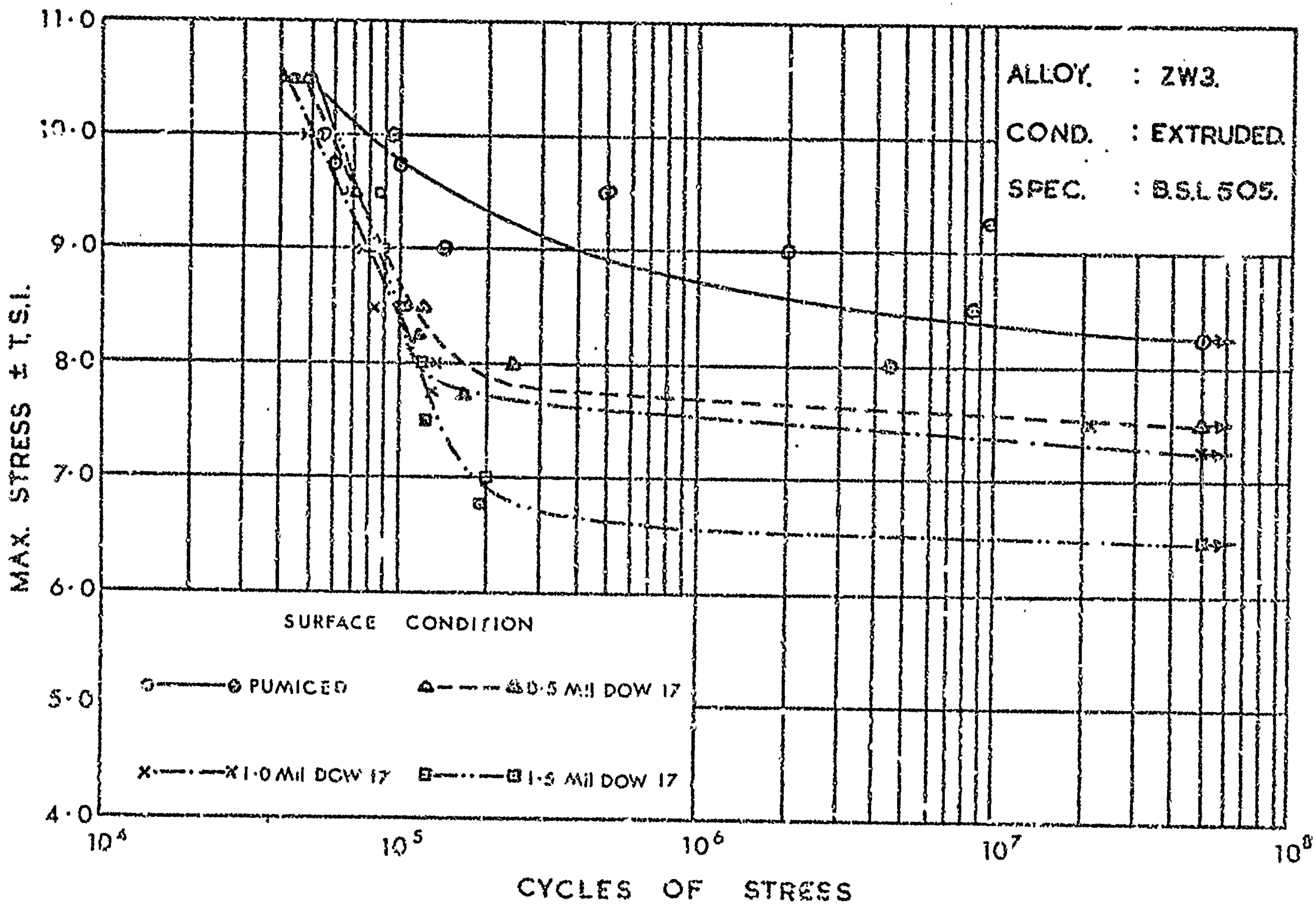


FIGURE 9 ROTATING BENDING FATIGUE TEST



10. APPENDICES

10.1 APPENDIX I - DETAILS OF SURFACE TREATMENTS

(1) CHROMATE TREATMENT TO DTD 911C

(a) Fluoride Anodising

Bath Composition	25% w/v Ammonium Bifluoride in water
Anodising Conditions (unless otherwise stated)	10 mins. at 120 volts (Ambient temperature)

(b) Stripping

Bath Composition	10% w/v Chromium Trioxide in water
Stripping Procedure	10 mins. immersion in boiling solution

(c) Activation

Bath Composition	15% HF in water
Procedure	5 minutes immersion at ambient temperature

(d) Chrome Manganese (Bath (v) of DTD 911C)

Bath Composition	10% w/v Sodium Dichromate Crystals
	5% w/v Manganese Sulphate ($MnSO_4 5H_2O$)
	5% w/v Magnesium Sulphate ($MgSO_4 7H_2O$)
Procedure	2 hours immersion at ambient temperature

(2) DOW 17 TREATMENT

Bath Composition	24% w/v Ammonium Bifluoride
	10% Sodium Dichromate Crystals
	9% v/v 85% Ortho-Phosphoric Acid
Current Density	30 amps/sq. ft. (unless otherwise stated)
Temperature	70 - 80°C
Time	Dependent on thickness required (See Fig. 1)

(3) H.A.E. TREATMENT

Bath Composition	12% Potassium Hydroxide 1.04% Aluminium 3.5% Trisodium Phosphate ($Na_3PO_4.12H_2O$) 3.5% Anhydrous Potassium Fluoride 2.2% Potassium Manganate
Current Density	60 amps/sq. ft. (85 volts max)
Temperature	Ambient
Time	90 minutes

(4) SURFACE SEALING

Material	CIBA Araldite 985E
Procedure	Samples preheated to 220°C - cooled to 60° and dipped. Air dried for 10 minutes, then stoved for 20 mins. at 220°C. Two further coats applied by dipping (inverting sample each time) and stoved for 20 and 45 minutes respectively at 220°C Total thickness of 3 coats: 1.5 mils

(5) J. HALLS CHROMATE PIGMENTED PRIMER

Material	J. Halls Chromate pigmented stoving epoxy primer Ref: 588/0066.
Application	2 sprayed coats stoved for 15 mins. at 125°C and 60 mins. at 200°C respectively. Total thickness approximately 1.0 mil

(6) OVERPAINTING TO DTD 5555

Material	Primer - Cellon chromate pigmented cold curing epoxy primer 2 parts SL5539: 1 part SL5538 + 10% Thinners TSL 5373 Finish - Cellon pigmented cold curing epoxy finish (White) 1 part SL5459: 1 part SL5538 + 10% Thinners TSL 5373
Application	1 Spray coat off each of primer and finish to give 1.0 mil and 1.5 mil coatings respectively.

(7) OVERPAINTING TO DTD 5580

Material	Primer - Pinchin Johnson's chromate pigmented epoxy primer SL6362 + Catalyst CSH 6331. Finish - Pinchin Johnson's White Pigmented Polyurethane Finish SL3054 + Catalyst CSL3055

10.2 APPENDIX II - TESTING PROCEDURES

(a) PHYSICAL TESTS

(1) Bend Test

A 4" x 2" x 16 swg. ZW3 panel was bent slowly round a $\frac{1}{4}$" diameter steel mandrel until fracture of the metal occurred. The sample was then removed carefully to maintain the 'hinge' formed by the coating system on the inside of the bend. The two fractured sections of the metal were then gently pulled apart. The extent of detachment of the coating system from the point of fracture indicated the adhesion. The performance of the coating on the inside and outside of the bend was noted.

(2) Shot Test

A 3" x 3" x 6 swg. ZW3 panel was used for the test. A 0.22 long rifle bullet was fired at the coated sample from a range of 25 yards. The extent of spalling of the coating system from the point of impact on the front and back faces of the panel was noted.

(b) CORROSION TESTS

(1) Seawater Spray-Humidity Test

This test was usually carried out on the panel from the shot test above. The panel was scribed with two crossed lines, 2 inches long, penetrating to the metal. Panels were then sprayed with a fine mist of natural seawater then exposed in a cabinet at 98 - 100% humidity for 6 months. Samples were resprayed after each intermediate examination.

The test was similarly conducted on flange assemblies, although no scribed damage was included.

(2) R.A.E. Seawater Spray Test

Panels were scribed with two, 2 inch long, crossed lines to expose the metal. They were then exposed in a shelter, open to the atmosphere on one side, and sprayed three times per working day with natural seawater, at Magnesium Elektron Ltd., Manchester.

The test was similarly conducted on flange assemblies, although no scribed damage was included, unless stated.

(3) Atmospheric Exposure Test

Panels or flange assemblies were exposed outdoors in the grounds of M.E.L. at Clifton Junction, Swinton for a period of 12 months or longer, depending on the degree of deterioration.

(4) Marine Atmospheric Exposure (Beaumaris)

Panels or flange assemblies were exposed outdoors in the grounds of Laird (Anglesey) Ltd., 200 yards from Mean Sea Level and 25 ft. above it. The site was used by the courtesy of Birmidal Developments Ltd., and Laird (Anglesey) Ltd.

(5) Distilled Water Column Test

A 5 ins. long column of distilled water contained in a glass tube was located on a flat area of the panel 1 ins. in diameter by means of rubber washers and clamps. The column was maintained in that position for up to 15 months with periodic testing and examining. Testing was carried out fortnightly by applying an e.m.f. of 12 v. between the base metal of the test panel and the top of the distilled water column, and noting any deflection of the microammeter in circuit. The deflection indicated the extent of conduction (water permeability) of the coating system.